# Particle Characterization

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**Module No. # 12**
**Lecture No. # 34**

## Practical Relevance of particle Characterization: Filtration

Welcome to the thirty-fourth lecture in our Particle Characterization course. In previous modules of this course, we have discussed various aspects of particle characterization, starting from morphological properties to interfacial properties, chemical properties, transport properties and so on. And then in the last module, we talked about some applications of particle characterization in nano technology. And in the last lecture, we discussed nano fluids which involve the addition of nano-particles to various fluids to enhance their properties.

In today’s lecture, we are going to continue our discussion of certain applications of particle technology, where characteristics of particles are very important. In particular, filtration is a technology that relies upon various properties of particles in order for it to work.

Filtration refers to the separation of impurities from a fluid stream. And the filter is then expected to retain these impurities that have been removed from the fluid, and make the impurities either available for analysis, or just use it as a way to collect these impurities and discard them.

When you talk about filtration, you can sub classify it as; particle filtration and molecular filtration. So, molecular filtration refers to removal of impurities in the form of organic and inorganic chemicals, and this is typically done by chemisorption or physisorption techniques.

On the other hand, removal of particles is predominantly a physical mechanism. And many of the transport properties of particles that we talked about earlier in this course such as; sieving, sedimentation, inertial impaction, interception, diffusion and electrostatic motion are actually made use of in order to remove particles effectively from a fluid gas stream.

Now, when we talk about filtration of particles, again you can classify it as; course filtration and fine filtration, or as free filtration and final filtration. Coarse filters are frequently encountered everywhere. So, things like fabric filters or granular bed filter are normally employed in microscopic applications like pollution control, for example, scrubbing of gases before they are release to the atmosphere. Another example of such techniques would be actually, even the electrostatic precipitators that are used in coal processing, for example, to remove ash particles.

So, these forces of electrical forces as well as simple capture by gravitational sedimentation or by interception are frequently resorted to remove large sized particles from gases and liquids. However, these methods are somewhat limited in their efficiencies when it comes to the finer particle sizes. So, as you start approaching micron and submicron sizes, these conventional methods of filtration that we are used to from everyday life really start to lose their effectiveness.

The two types of filters that are most widely used for removing the finer particle from fluids are; fibrous filters and membrane filters. Now of the two, fibrous filters are considered to be more effective for the coarser regime of the fine particles size range. So, the final filtration, that is designed to remove particles in the submicron to nano sizes, is typically done using membrane filtration.

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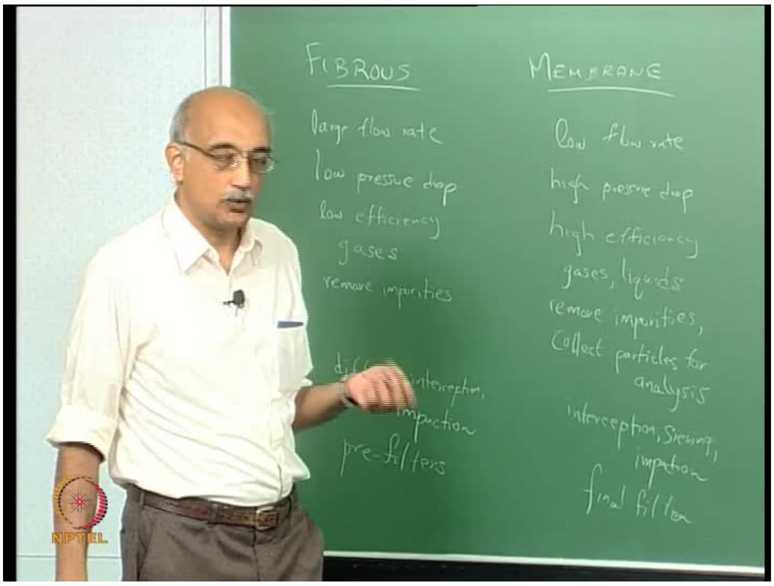

So, if you want to look at the differences between fibrous filters and membrane filters; fibrous filters usually have large flow rates and relatively low pressure drop whereas, membrane filters typically have low flow rate and relatively high pressure drop. So, it essentially means that the fibrous filters also have low efficiency comparatively speaking and the fibrous filters have high efficiency. Actually, the fibrous filters can be used only for gases; whereas, the membrane filters can be used for gases as well as liquids. And the primary purpose of a fibrous filter is to remove impurities whereas, the primary purpose of a membrane filter is to remove impurities as well as collect particles for analysis. In terms of particle collection mechanisms, fibrous filters rely upon diffusion, interception and impaction whereas, membrane filters rely upon mainly interception, sieving and impaction.

The reason being, that a fibrous filter by definition has depth, and so, the diffusion becomes ineffective mechanism because particles have a long resonance time as they go through the filter medium. So, they can follow a tortuous pathway and the likelihood that they will get removed during their diffusional transport is very high. Whereas, membrane filters are usually very thin and so, diffusion is not a very effective mechanism for removing particles, because the pathway through the membrane is just not long enough and so, you tend rely more on interception and impaction as the primary mechanisms of removing particles.

So, again the fibrous filters would be more conventionally used as pre-filters whereas, the membrane filters would be the absolute or final filter that you employ at point of views, before you introduce the chemical, whether it is a gas or a liquid into your process.

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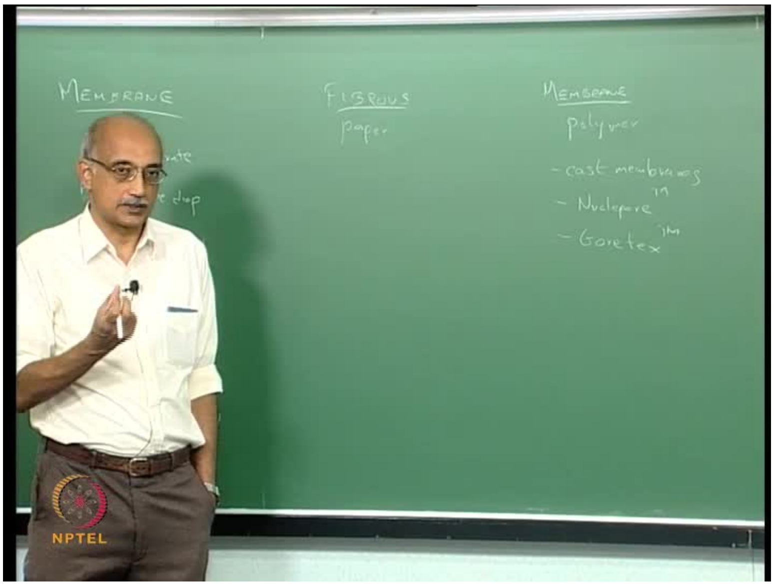

So, again let us look at some more differences between fibrous and membrane. Fibrous filters are basically made of a paper, and membrane filters are made of polymer. So, the way this basically works, to make a membrane filter, you typically take the shavings from a paper cutting machine, so you take these loose fibers that were in a very bulky low density form, and you make a slurry of these fibers in a liquid with a binder material. And then you essentially flow this slurry over a screen, and you allow the solvent to evaporate. As the solvent evaporates, you essentially form one layer of a porous material, which is then dried and then stored in the form of rolls.

So, these fibrous filters are available to you in these large roll forms and they are essentially porous sheets of paper. They typically have about 10 percent solids fraction or 90 percent void fraction. And the fibrous filters have a tendency to shed, because they are essentially made of these fibers that have been bound together. So, there is always a chance that once in a while, some fibers could actually be contributed by the filter itself, and that is the reason you do not use them as a final filter, because there is a potential that the fiber itself can become a contaminer. And so, the fibrous filters are quite good.

One of the big advantages is as they filter more material their efficiency actually increases, because they simply add to the depth of the fiber bed, and that is why pressure drop is really not in issue with the fibrous filters.

Membrane filters, on the other hand, are essentially thin sheets of polymers with holes in them. Now, there are really three types of membrane filters; cast membrane filters, nuclepore filters and Gore- Tex filters. These are trade mark names and nuclepore is marketed by a company called Millipore, Gore- Tex is marketed by a company called W. L. Gore.

The way that the membranous is made is different in each case. In a cast membrane filter, you essentially take the polymer, and you completely dissolve it in a solvent, and cast it in sheet form. So, the process is actually very similar to the casting of a fibrous filter. And here again, as the cast material dries, it leaves behind a layer or a sheet of polymer with porosities in it.

A nuclepore filter is one, where there is much more control over the size as well as the placement of this pores. Essentially in this case, you take polycarbonate polymer sheet and expose it to nuclear radiation in a nuclear reactor. And then, you inspect the polymer film for damaged areas and you etch out these damage sides. So, when you do that essentially, you get a sheet with discrete circular holes in it. And these holes are very easily distinguishable from the flat part of the material. So, the nuclepore filters are very useful both for filtering out particles and also collecting particles for analysis, because the particles will be retained at these hole locations, and they will be fairly conveniently analyzed, when you put it in a microscope, particularly a scanning electron microscope or a TEM.

Gore-Tex on the other hand, is basically a Teflon type of polymer material and you just keep stretching it, just keep pulling it till it starts rupturing and forming pores in it, and that is basically how a Gore-Tex filter is made. The Gore-Tex material is relatively expensive compare to the other two, but very clean, one of the big advantages of Teflon is that, it is a very cleanable material. The nuclepore is most widely used particularly for collecting particles for analysis, because of the way the uniformity of the holes that are etched in the polymer material. And then, the cast membranes are probably the lowest cost bulk manufacturing method for membrane filters.

So, again the key point is fibrous filters are effective, but have limitations in terms of delivering an absolute final clinginess of the fluid. The membrane filters in that sense, are much better, much more precise, but on the other hand, they have to be replaced more frequently, because they tend to clogged as a particles are collected on the surface.

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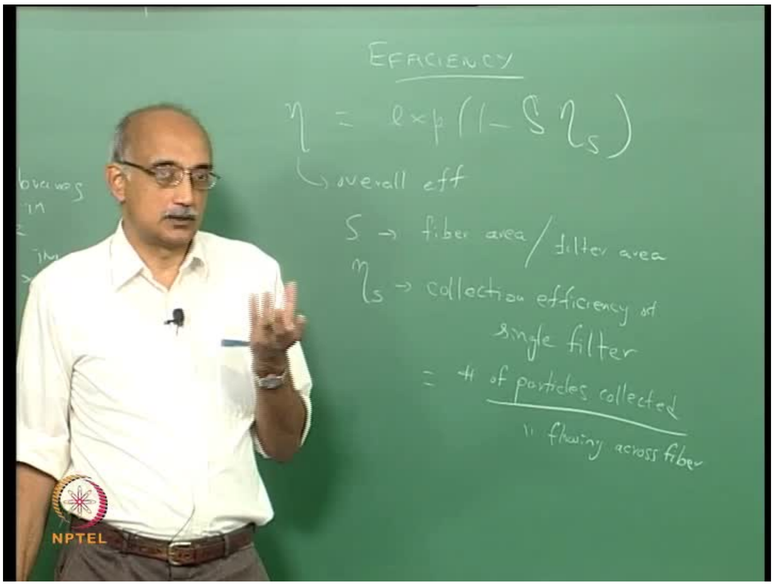

The efficiency of a filter is; obviously, an important parameter. In the case of a fibrous filter, the overall efficiency eta can be calculated as exponential of 1 minus S times eta s; where eta is the overall efficiency, S is the fiber area per unit area of filter, that is what fraction of the total area of the filter medium is actually the area of the fiber itself, and eta s is the collection efficiency of a single filter. So, this is basically equal to the number of particles collected to number of particles flowing across the fiber. So, this is like capture efficiency that we had talked about earlier, in the context of surface interactions with particles. So, if you look at this expression the collection efficiency of a single filter as well as the total area of fiber in the filter are obviously important. And the exponential relationship comes about, because as you add depth to the filter, the filter efficiency keeps increasing, and essentially there is an exponential drop in the number of particles that can get through with the addition of the area to the filter as well as with the increase in efficiency of a single fiber.

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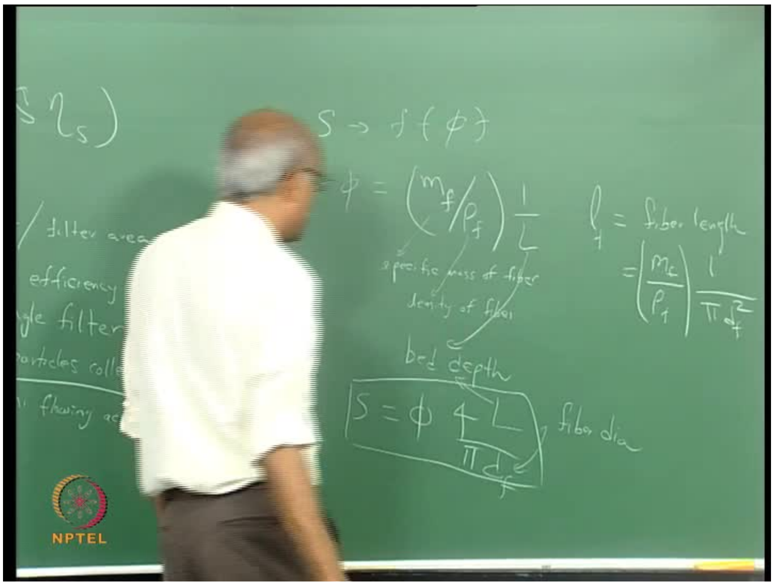

Now, the definitions of S and eta s are obviously important. Now, S which is the specific surface area of fiber in the material is related to the volume fraction of the fiber in the material. So essentially, the higher the solids fraction, it is also called the packing density of the fiber in the filter matrix, the higher will be the value of S. Now, this term volume fraction of the solid material in the matrix can be taken as m p or m f over rho f divided by 1 over L; where m f is the specific mass of the fiber material, that is mass of fiber per unit area of packing, rho f of course, is the density of the fiber and L is the bed depth, the total depth of the filter bed that the particles have to traverse through.

So, when you relate S and the solids fraction, the expression essentially goes like this; S equals 5 times 4 times L over phi d f; where L again is the depth of the bed and d f is the fiber diameter. And the reason that this is, is because you can actually do the calculation, you can define a specific length of the fiber as l f, which is the specific fiber length, which will be equal to m f over rho f times 1 over pi times d f squared, and from that you can essentially calculate or estimate this expression.

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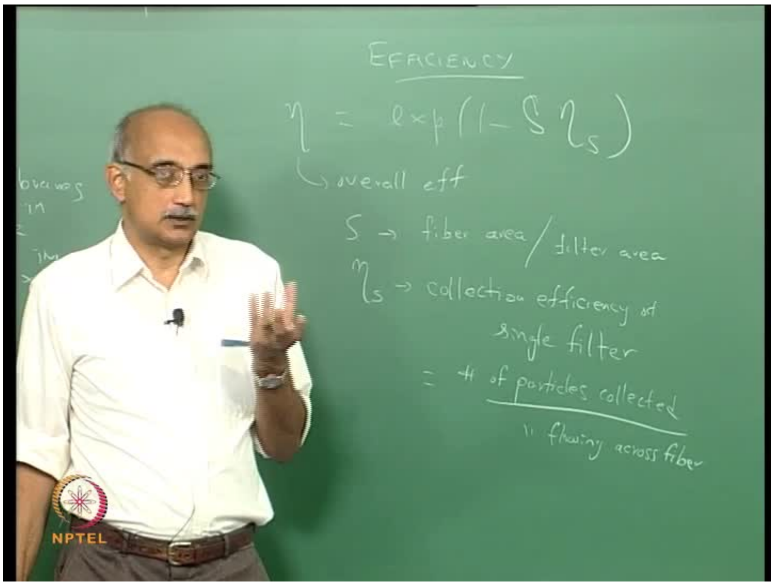

So, once you know, really three parameters; the depth of the bed, the diameter of the fiber and the solids fraction in the fiber material, you can calculate the S value, which is the specific surface area of the filter. And so, once you know S and if you know eta s, you can calculate the overall filtration efficiency. But the challenge obviously, is in calculating this. How do you calculate the collection efficiency of a single filter? Now here, we go back to the material that we have covered earlier in this course. This basically depends on transport properties of particles as well as the adhesion properties to surfaces, in this case the surface of the fiber that the particle is flowing across.

Before we do that, the other parameter that we are interested in is a pressure drop. As you know in filtration technology, virtually anything you do to improve efficiency also results in a higher pressure drop, and you always have to balance between the two. So, in the case of a fibrous filter, if you look at the expression for pressure drop, delta p for a fibrous filter can be related to the drag force multiplied by the specific length of a fiber.

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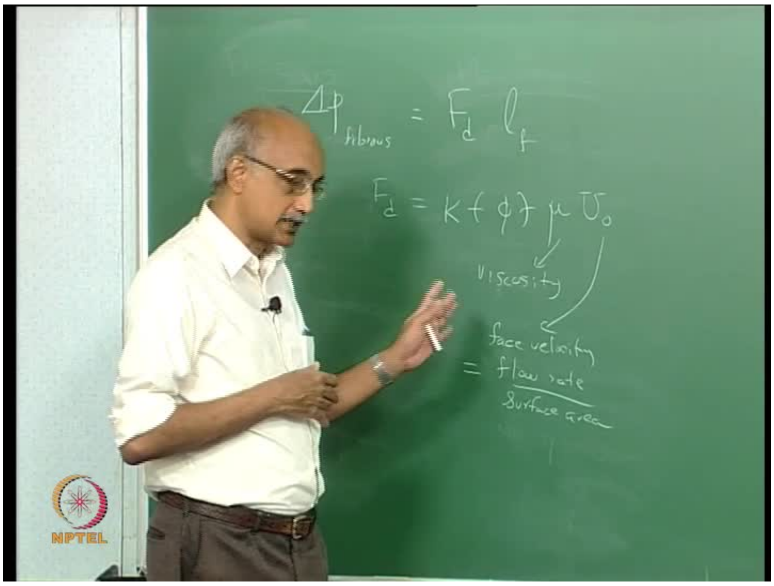

Now, this parameter f d, the drag force itself is some constant, which is a function of the volume fraction times mu times U 0; where mu is the viscosity of the fluid and U 0 is the face velocity, which is defined as flow rate divided by the surface area of the filter. And so, essentially the higher the flow rate that the filter has to experience, the greater will be the pressure drop. And also the greater the viscosity of the fluid that is being handled, the higher will be the pressure drop. And finally, the solids fractions, the higher the solids fraction, the greater the filtration efficiency, but the greater the pressure drop.

So, again there are some optimization exercises you can go through. If you want to get maximum filter efficiency at minimum pressure drop, either you have to make some compromises or you have to be very clever in the way you design the fiber filter, in order to minimize pressure drop, but still keep a high efficiency. And actually you can see from this expression that, filtration of liquids is going to be a huge challenge primarily because of the viscosity, the higher viscosity will lead to higher pressure drop. So, the filtration of liquids, it is a very different challenge, and we will see later that it is not only because of this, but even some of the transport mechanisms that are used to capture particles are very different, in the case of liquids. Some of the mechanisms that are present when filtering gases, are absent when you are trying to filter liquids, and that presents a challenge as well.

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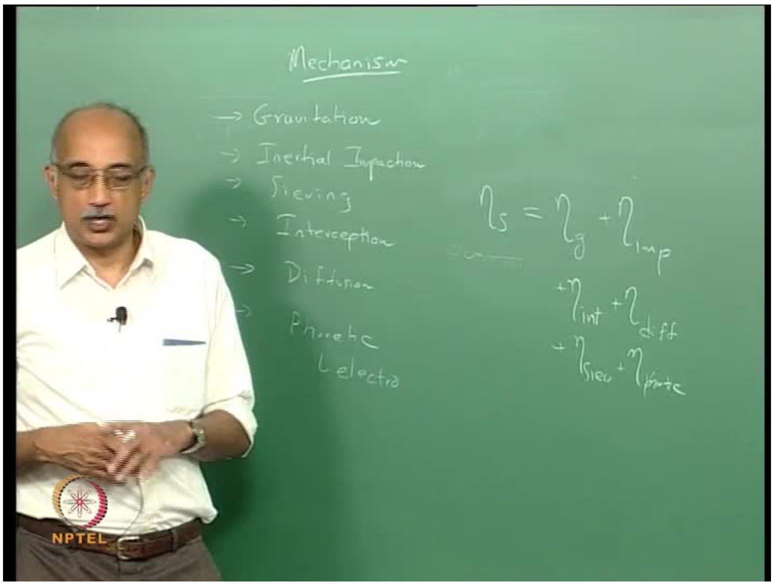

Now, let us look at some of the mechanisms of particle capture, and here is where obviously, the particle characteristics play a huge role. The primary mechanisms of particle capture, if you go from a large size to a small size, for very large particles gravitation, sedimentation is a method for removing particles by filtration. So, for example, that granular bed or the fabric filter that we talked about earlier, essentially work by a gravitation mechanism, particle simply settle into the filter media and get removed in that fashion. But that method is only valid for particles that are in the millimeter and larger size range, for particles smaller than that, gravitation is just not a large enough force.

So, the next one would be inertial impaction. As we have seen earlier, inertial impaction is a mechanism that has particular relevance for particles that have stokes numbers larger than about 0.5, and they show a significant deviation from the stream lines of fluid flow. So, the particles initially separate from the fluid and this separation can then be used by the filter medium to capture the particles. So, the capture efficiency, both in gravitation as well as inertial impaction, is quite high, close to 1. And but again, they are primarily effective only for the larger sizes.

The next method would be sieving and interception, which are actually very similar mechanisms of particle capture. So in both, essentially the particle will approach a hole, and if the particle is larger than the hole, it gets retained, if it is smaller, it passes through. So, very simple in concept. Interception again, is very similar, in the sense that, here the particle is not approaching the collection surface in a vertical direction, but in a parallel direction. And again, as we have seen before, you essentially have a surface like this, and if the particle is flowing in such a way that its stream line carries it very close to the surface, such that it comes in contact with the surface, then the particle will preferentially detach itself from the fluid and attach itself to the surface.

So, interception again differs from inertial impaction in one main point, that the particles are not deviating from stream lines, they are still following fluid stream lines, but the stream line flow is such that, it brings the particle in close proximity to the surface, and therefore, the particle gets attached to the surface and gets removed.

The next mechanism would be diffusion. And of course, the diffusion will become more and more dominant as the size decreases. So, you can imagine that, as you start approaching submicron and nano dimensions, diffusional forces will start to take over. So in fact, with fibrous filter and sub-micron particles, you have virtually 100 percent removal efficiency. However, with the membrane filter, the very fine particles can certainly escape, particularly if they are highly non-spherical in shape. Because membrane filters rely upon, essentially an interception mechanism. You have circular holes and if the particle is not circular in its profile, for example, if its needle like, you can easily penetrate through, and that is why you cannot simply rely on membrane filters to give you final product clinginess, you have to combine fibrous and membrane filtration, in order to leverage various transport properties of particles and remove all of them over a very wide size range.

Of course, the phoretic forces are also important in collecting particles. For example, you can have electro phoretic filters, and the electrostatic filters can be both active and passive. What you mean by an active electrostatic filter is, where the filter medium is deliberately charged to a certain charge, and this is particularly useful in removing charged particles of the opposite charge, obviously. Whereas, a passive filter, passive electrostatic filter is simply one that develops a charge on its own. For example, wool, wool is a material that will develop static charge quite easily, simply by tribology and the charge would be sufficient to enhance the capture efficiency. And there are also polymers called electrets that can be used to develop a charge over time and remove particles using that mechanism.

So, the overall, if you look at eta s, which is the particle removal efficiency of a single fiber, it is obviously a combination of all this; the efficiency due to gravitation plus efficiency due to impaction plus efficiency due to interception plus due to diffusion plus sieving plus phoretic and so on. And obviously, for the eta s value is going to be very size dependent, for very fine particles diffusion and electrostatic effects may be predominant, for as very large particles gravitation and impaction may be predominant. But in general, the overall single fiber collection efficiency is going to be a sum of all these individual efficiencies.

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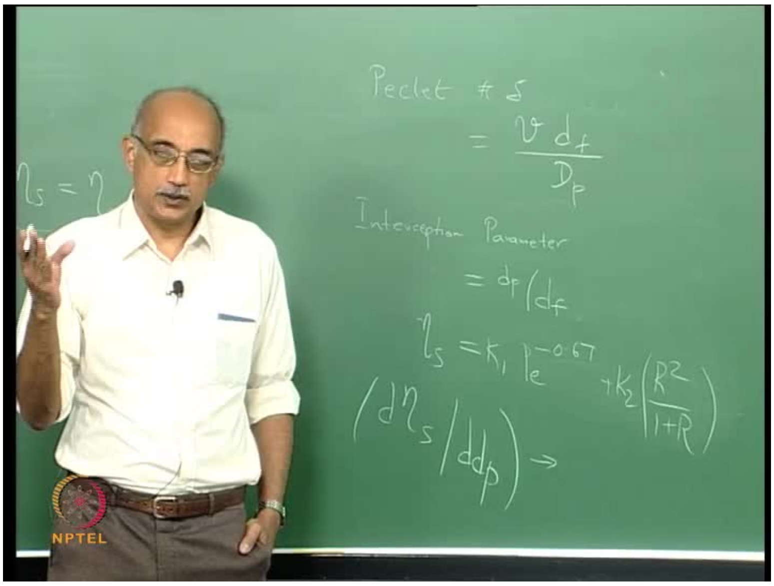

Now, as we have seen before, many of these mechanisms; gravitation, impaction, diffusion, phoresis, can all be represented in terms of Peclet number. Peclet numbers are ratio of the characteristic velocity that is associated with that particular mechanism times a characteristic length, which in this case would be the fiber diameter divided by, you can use for example, the diffusivity of the particle as your reference value in the denominator. So, you can define these Peclet numbers for each of these mechanisms. Interception on the other hand, really cannot be represented in terms of a Peclet number, instead use something called an interception parameter, which is the ratio of d p over d f, that is the ratio of the particle that is approaching a fiber to the diameter of the fiber itself. And, the efficiency of filtration can then be expressed for a single fiber as a constant K 1 times the Peclet number to the power minus 0.67 plus K 2 times R squared by 1 plus R.

So, this is a semi empirical formula that is used to estimate the efficiency of a single fiber. So, essentially as the lower the Peclet number, the higher will be the associated filtration efficiency, and the dependence of the interception parameter goes as R square over 1 plus R. So, this is the formula that needs to be plugged into the overall filtration efficiency of a fibrous filter, eta equals exponential of 1 minus eta s times s.

If you look at this parameter, it is obvious that if you take d eta of a single filter over the particle diameter, if you take the differential of the filtration efficiency as a function of the particle diameter, this will have, and then you plot this versus, actually you plot d p versus eta s, if you plot the particle diameter versus the collection efficiency, it is clear that it will have a non-monotonic behavior, it is not going to have a simple monotonic dependence.

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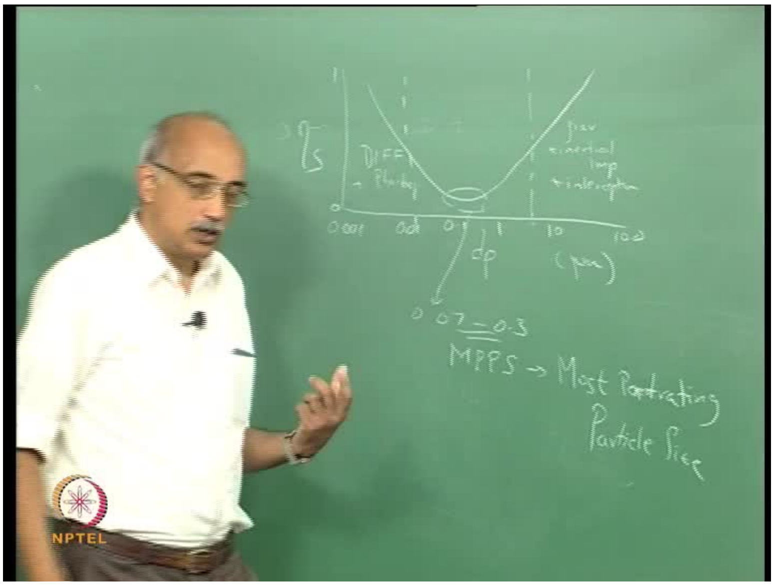

And in fact, a lot of analysis has been done, a lot of experiments have been conducted to study this more closely. And what has been found is that, if you plot d p versus eta s, and you go from 0.001 microns to 0.01 microns 0.1 1 10 100, and on this axis you go from 0 to 1, the behavior is always the same, no matter what the material of the filter, no matter what the material that you are you are filtering, there is a universally valid trend that you can see and it basically looks like this. The filtration efficiency is very high in the smaller sizes because in this range diffusion plus your phoretic effects dominate. So, they result in very high filtration efficiency whereas, in this range, gravity plus inertial impaction plus interception are first order effects and they again lead to very high filtration efficiencies.

So, the minimum in the filtration efficiency is obtained in a size range that is between 0.07 microns to about 0.3 microns. And in fact, this size range is known as the MPPS range, which stands for Most Penetrating Particle Size. Because filtration efficiencies in this size range are very poor, the likelihood of a particle penetrating the filter is maximum in this size range as well. And that is why it is called the most penetrating particles size range. So, what that means is that, particles in this size range have the highest probability of escaping the filter and getting into your process. So, if you are trying to access the efficiency of the filter, then you should really challenge it, not with particles in this range or with particles in this size range, but rather with particles in this size range. Because then, you will really be testing the capabilities of the filter.

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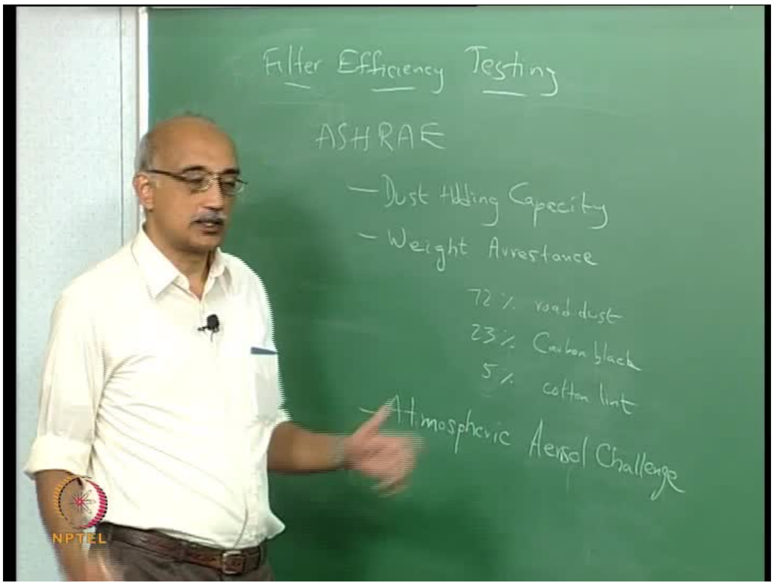

And in fact, filter efficiency characterization is something that is; obviously, of a lot of importance in many manufacturing industries. So, filter efficiency testing is done very professionally by certified organizations. The one organization that is very active in this area is ASHRAE, which stands for American Society of Heating Refrigeration and Air conditioning Engineering. ASHRAE has actually set the standards for measuring the efficiencies of particulate filters. The measurement technology itself is pretty simple, you essentially challenge the filter with some impurity, and you measure upstream and downstream concentrations of the impurity, and the drop in concentration is a reflection of the effectiveness or efficiency of the filter. The challenge really comes in deciding what is the challenge material, what do you use to access the efficiency of the filter.

Some of the methods that ASHRAE has defined; first one is called the dust holding capacity, and a related test is weight arrestance. Now, these test simply use a mixture of 72 percent road dust, 23 percent carbon black and 5 percent cotton lint, as the challenge material. So, it is a mixture of common environmental contaminants that you find, its mostly dust to represent the inorganic fraction, the carbon black represents the organic fraction of the environmental contamination and cotton length stands for all the fibrous materials that are emitted by garments and fabrics and so on.

There is also a test called the Atmospheric Aerosol Challenge. Here, the material that you use to flow through the filter is simply the ambient air. So, just take the air from the environment and push it through the filter and you measure the solids concentration in air, before and after the filtration to assess the effectiveness of the filter.

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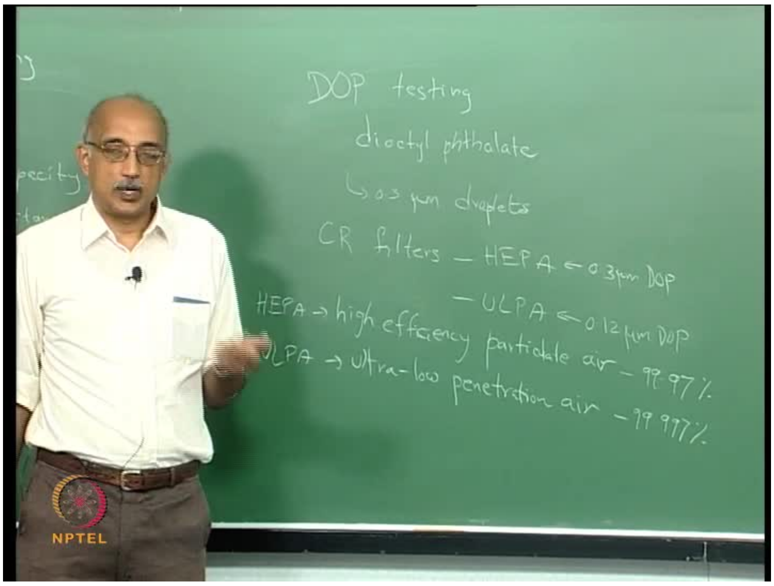

So, these methods, I mean this one is obviously for large particles, this one is for particles in a wider size range. But if you want to challenge the filter in the MPTS size range that I talked about earlier, then you have to specially formulate that material, and that method is known as DOP testing, DOP stands for Dioctyl phthalate. So, Dioctyl phthalate is first taken in liquid form, the liquid is evaporated and then re-condensed to form very fine nuclear. As we have seen earlier, when you evaporate a substance and then recondense it, you form small nuclei that are of the order of nanometers and eventually growing to submicron sizes. So, in this technique, you can actually control the resulting aerosol to a very uniform mono dispersed size distribution that is centered around 0.3 microns. So, from this you essentially get 0.3 micron sized droplets of DOP, which you then challenge your filter with.

When you start characterizing filters at this level, these filters are typically used for clean room applications, where it is very critical to maintain particulate clinginess of the environment. And there are really two types of clean room filters; HEPA filters and ULPA filters. HEPA stands for High Efficiency Particulate Air filters and ULPA stands for Ultra-Low Penetration Air filters.

So, the HEPA filters are challenged using 0.3 micron DOP droplets that have been generated using this technique. ULPA filters are challenged using 0.12 micron DOP. And the requirement for a HEPA filter is that, it must be able to remove 0.3 micron sized DOP droplets with an efficiency of 99.97 percent. So, in order for a filter to qualify as a HEPA filter, which is used in clean rooms, it must be able to demonstrate 99.97 percent removal of DOP at 0.3 micron.

The requirement for the ULPA filter is even more stringent. It has to be able to show 99.997 percent removal of 0.12 micron sized DOP and so, this is the ULPA filter is used for a higher class of clean room compare to HEPA filter. HEPA filter is used for clean rooms up to a class 100 or so, whereas ULPA filters are required, when you start approaching class 10 class 1 clean rooms. As you may call from our discussion of clean room classes, the clean room class refers to the number of particles per unit volume of the clean room air, and as that number drops, the clinginess requirements become more and more stringent, and you have to resort to techniques like ULPA filtration, in order to be able to achieve those standards.

Now, when we talk about filtration in a clean room, we are mainly talking about filtration of gases. And as I mentioned earlier, gases are filtered using less complicated filters than liquids because the demands are less. Liquids are much more difficult to handle, I mean for example, fibers would not even work, fibers will simply either dissolve or swell in liquids.

So, a fibrous filter really is only suitable for filtering of gases. When you talk about filtering of liquids, the only resort you have is to use membrane type filters. So, if you look at the mechanisms that we have outlined earlier and think about which of those are really applicable in the case of liquids, a mechanism like sieving still works reasonably well, I mean you can use a liquid sieve and sieve particles.

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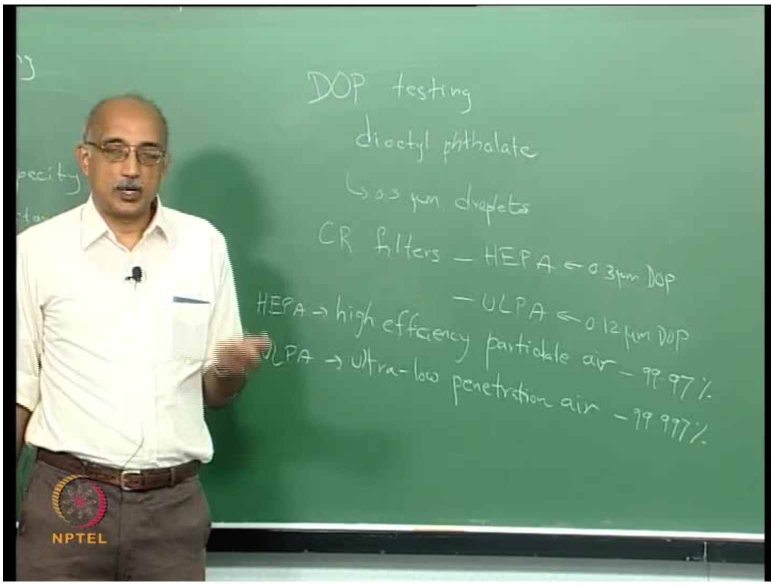

But a mechanism like, for example, diffusion. Particles are not able to diffuse very fast in liquids because of the high viscosity, but that can be counteracted to some extend by the fact that, in liquids the particles have a longer resonance time, for the same reason, because of the higher viscosity they spend more time in the liquid medium. So, the probability of capture is a little higher.

Another mechanism is interception. In the case of interception, the mechanism is virtually the same for both filtration of gases and liquids. Inertial impaction on the other hand, is very poor in liquids. Because particle really cannot impact on a surface when it is suspended in liquid, it can happen very easily in a gas, but again the viscosity of the liquid layer will essentially prevent the particle from approaching the surface at high velocities.

And then, you have electrostatic forces. Electrostatics, as we have seen before, water is a good medium for disposing of static charges and the field strength is significantly reduced. So, the particle to surface adhesion, when it is immersed in a liquid is much lower compared to in a dry condition. However, there are the double layer forces that develop in liquids and it may be possible that they can assist in some way with collection of particles. But electrostatic forces are much more effective again in filtering of gases.

So, for all these reasons the filtration of liquids is usually much more challenging than filtration of gases, the efficiencies are lower, pressure drops are higher, cost is higher. So, the filtration of liquids is typically done only to the extent necessary. It is not something you do just for the sake of buying an insurance, basically, it is something you do when there is a demonstrated need to do it.

The other problem with many liquids is their chemically reactive nature. Many of the filter media will actually be attacked by when you try to filter liquids and so, you are force to use something like, Teflon, the Gore-Tex material is very good for filtering of particles from a liquid, but the cost is very high. Teflon itself is a an expensive polymer compared to the polymers that are typically used in cast membrane filters or even there the nucleopore filters.

So, the thing with filtration technology is the particle characteristics obviously, play a huge role, again particle size is the most important parameter. The filtration methodology you use is very much dependent on the size range that you are trying to filter. Size and shape also plays an important role, because many of these mechanisms are effective really, only assuming a spherical or near spherical particle shaped distribution. If you have a population of particles with highly non-spherical, highly elongated shapes, many of these mechanisms may not be effective. I mean, obviously diffusion would be even more effective for non-spherical particles, but mechanisms like sieving and interception probably will lose their effectiveness and the particles become more non-spherical.

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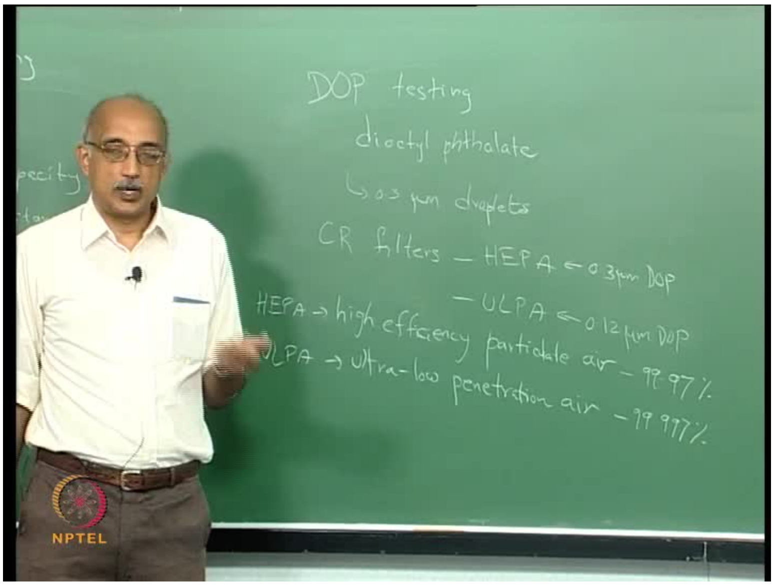

The chemical nature of the particle can play a role, but it is most more likely to happen in liquid media and less likely to happen, when you are trying to filter gases. And of course, things like mass of the particle, density of the of the particle all play some role, but the transport properties or the most critical aspects in filtration and filter design.

The transport characteristics of the particles, pretty much decides whether a filtration system is going to work for you or not, and that is really what you also make use of when you are trying to optimize the design of your filtration system. Again it is a constrained optimization problem, as a chemical engineer we are used to those, essentially you are trying to maximize your filtration efficiency, but with the constrained that cost goes inversely as filtration efficiency. So, these are thing that you really have to keep balancing as a process engineer. And a good understanding of particle characteristics in all these respects, will certainly help you in terms of this optimization exercise.

So, let us stop this lecture at this point. In the next lecture, we will talk the little bit more about the particle characteristics that are of particular importance in deciding the yield and reliability of microelectronic devices, and how we can actually leverage some of these characteristics, in order to again maximize the yield in reliability of microelectronic products. Any questions on what we discuss today? See you at the next lecture then.